

(PCT Rule 61.2)

To:

in its capacity as elected Office

1. The designated Office is hereby notified of its election made:

☒ in the demand filed with the International Preliminary Examining Authority on:

21 August 2000 (21.08.00)

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PATENT COOPERATION TREATY

PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

09/890917

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Applicant's or agent's file reference PCT/2000/031		FOR FURTHER ACTION See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)
International application No. PCT/ZA00/00019	International filing date (day/month/year) 08/02/2000	Priority date (day/month/year) 08/02/1999
International Patent Classification (IPC) or national classification and IPC C08L97/02		
Applicant SAPPI LIMITED et al		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.


2. This REPORT consists of a total of 5 sheets, including this cover sheet.

- ☒ This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

These annexes consist of a total of 17 sheets.

3. This report contains indications relating to the following items:

- I ☒ Basis of the report
- II ☐ Priority
- III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- IV ☐ Lack of unity of invention
- V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- VI ☐ Certain documents cited
- VII ☐ Certain defects in the international application
- VIII ☐ Certain observations on the international application

Date of submission of the demand 21/08/2000	Date of completion of this report 18.05.2001
Name and mailing address of the international preliminary examining authority:  European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465	Authorized officer Ehrenreich, W Telephone No. +49 89 2399 8675



INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/ZA00/00019

I. Basis of the report

1. With regard to the **elements** of the international application (*Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17)*):

Description, pages:

1-13 as received on 16/03/2001 with letter of 15/03/2001

Claims, No.:

1-16 as received on 16/03/2001 with letter of 15/03/2001

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language: , which is:

- ☐ the language of a translation furnished for the purposes of the international search (under Rule 23.1(b)).
- ☐ the language of publication of the international application (under Rule 48.3(b)).
- ☐ the language of a translation furnished for the purposes of international preliminary examination (under Rule 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

- ☐ contained in the international application in written form.
- ☐ filed together with the international application in computer readable form.
- ☐ furnished subsequently to this Authority in written form.
- ☐ furnished subsequently to this Authority in computer readable form.
- ☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
- ☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. The amendments have resulted in the cancellation of:

- ☐ the description, pages:
- ☐ the claims, Nos.:
- ☐ the drawings, sheets:

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

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(Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)

6. Additional observations, if necessary:

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes:	Claims	1-12,14,15
	No:	Claims	13,16
Inventive step (IS)	Yes:	Claims	1-12,14,15
	No:	Claims	
Industrial applicability (IA)	Yes:	Claims	1-16
	No:	Claims	

2. Citations and explanations see separate sheet

Ad Item V

Novelty, Art. 33(2):

None of the prior art documents disclose the binding mixture claimed including

- urea-formaldehyde (UF) resin
- MDI
- one or more catalysts selected from a) or b) as claimed in claim 1.

Therefore, the binding mixture claimed in claims 1-12, the process for preparing chipboard using the above binding mixture according to claim 14 and 15 and the chipboard of claim 16, as far as the claim refers back to claims 1-12,14,15, are novel.

The wording of claim 13 "an additive for a UF binding mixture for use ... the additive including ... MDI and a catalyst ..." has to be understood such that the MDI/catalyst mixture according to original claim 1 is claimed because features like "for use as" or "suitable for" merely define a result to be achieved and do not limit the scope of a product claim.

The subject-matter of claim 13 is therefore not novel vis à vis D1 (WO-A 94/05475, claims 1-3,5-10 in context with p. 4, second para to p. 7, second para and the example of p. 8), D2 (EP-A 0 346 059, claims 1,5; p. 3, ll. 10-22 and examples 9,10), D3 (EP-A 0 039 137, claims 2,3,8 in context with p. 4, ll. 4-30). The same applies to claim 16 as far it refers back to claim 13.

Inventive step, Art. 33(3):

Closest prior art is D1 disclosing a binding mixture for use in manufacturing chipboard, the mixture including an effective amount of MDI and one or more polyurethane catalysts selected from tertiary amines, hydroxyamines or ether amines. The MDI may be water-emulsifiable and the catalyst may be a delayed-action catalyst. D1 also discloses a process for manufacturing chipboard, the process including adding to or using in a binding mixture an effective amount of MDI and a catalyst as mentioned above and mixing the binding mixture with wood particles.

The subject-matter claimed differs therefrom in that UF resin has in addition been used. There is no indication either in D1 or in D2/D3 that would motivate a person skilled in the art to add UF resins to the above MDI/catalyst binding mixture in order to produce

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT - SEPARATE SHEET**

International application No. PCT/ZA00/00019

chipboard. Thus, the subject-matter of claims 1-12, 14, 15 is based on an inventive step.

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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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(21) International Application Number: PCT/ZA00/00019 (22) International Filing Date: 8 February 2000 (08.02.00) (30) Priority Data: 99/0969 8 February 1999 (08.02.99) ZA (71) Applicant (for all designated States except US): SAPPI LIMITED [ZA/ZA]; 6th Floor, Sappi House, 48 Ameshof Street, Braamfontein, 2017 Johannesburg (ZA). (72) Inventor; and (75) Inventor/Applicant (for US only): MABOKA, Sydney [ZA/ZA]; 20 Cathedral Street, 6529 George (ZA). (74) Agent: D.M. KISCH INC.; P.O. Box 781218, 2146 Sandton (ZA).		(81) Designated States: AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG). Published <i>With international search report.</i>	
(54) Title: TIMBER PROCESS AND PRODUCT			
(57) Abstract <p>This invention provides a binding mixture for use in manufacturing chipboard, the mixture including an effective amount of methyl di-isocyanate ("MDI") and one or more polyurethane catalyst(s) being selected from the following: (a) one or more amine compound(s) including aliphatic and aromatic tertiary amine derivatives of phenols, esters, ethers, alkenes and/or alcohols; or (b) one or more organometallic compounds of tin, bismuth, zinc, iron, and/or alkali metal salt(s); or (c) suitable mixtures of (a) and (b) above.</p>			

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TIMBER PROCESS AND PRODUCT

5 INTRODUCTION

This invention relates to a timber process and product. More particularly this invention relates to a process for manufacturing so-called particle board or chipboard, and to a timber product in the form of such particle
10 board or chipboard when manufactured by or from the aforementioned process.

BACKGROUND TO THE INVENTION

- 15 It is known in the manufacture of particle board or chipboard (hereinafter referred to as "chipboard") that a urea-formaldehyde resin (hereinafter referred to as "UF") is used as a binder or adhesive to bond the particles or chips of wood.
- 20 Typically such binders contain approximately 55% to 60% formaldehyde (in moles) but because of recent awareness of the health problems associated with the use of formaldehyde, the amount of formaldehyde in such resins is generally being reduced. Hence the amount of formaldehyde in the aforementioned resins has been reduced to
25 approximately 50% or less (in moles).

The aforementioned resins need to be cured, and the curing process is accelerated by heating in a press under pressure i.e. heat is applied by hot metal platens on both sides of a so-called mat of glued chips. Such chipboard pressing takes place either in a batch-type (using daylight
5 presses) or in a continuous process i.e. using continuous (for example roller) presses.

It is also known in the chipboard industry that methyl di-isocyanate
10 (hereinafter referred to as "MDI") may be added to the aforementioned type of resin to hasten the curing process i.e. to effectively increase the speed of the curing process and hence to reduce the curing time. However, because of the high cost of MDI and the amount required to effectively hasten the curing process, the use of MDI is not cost-
15 effective on an industrial scale.

OBJECTS OF THE INVENTION

It is accordingly a general object of the present invention to provide an
20 improved binding mixture for chipboard manufacture.

It is also an object of the present invention to provide an improved process in which such improved mixture is used, which is both cost-effective and which increases the efficiency and rate of curing of the binding mixture.

5

It is a further object of the invention that the aforementioned binding mixture and process will lead to an increase in productivity or production flowing from shorter curing periods resulting from use of the aforementioned binding mixture.

10

SUMMARY OF THE INVENTION

According to one aspect of the present invention, there is provided a binding mixture for use in manufacturing chipboard, the mixture including
15 an effective amount of MDI and one or more polyurethane catalyst(s) being selected from the following:

- (a) one or more amine compound(s) including aliphatic and aromatic tertiary amine derivatives of phenols, esters, ethers, alkenes and/or alcohols; or
- 20 (b) one or more organometallic compound(s) of tin, bismuth, zinc, iron and/or alkali metal salt(s); or
- (c) suitable mixtures of (a) and (b) above.

The MDI may or may not be water-emulsifiable, and the or each polyurethane catalyst(s) may or may not be (a) delayed-action catalyst(s).

- 5 To the mixture may be added an effective amount of toluene diisocyanate ("TDI") and/or one or more internal wetting and release agents.

The aforementioned mixture(s) may of course include a suitable amount
10 of urea-formaldehyde resin which may or may not have suitable quantities of polyol(s) added thereto.

The amount of formaldehyde in the aforementioned urea-formaldehyde resin may preferably be approximately 50% (in moles) i.e. may be less
15 than, equal to, or more than 50% (in moles).

By using a catalyst as aforesaid, the effective amount of MDI required is reduced relative to the amounts known in the prior art to be sufficient to hasten the curing process of the mixture.

20

Suitable amines or amine compounds may be selected from the following:

2-dimethyl ethanolamine (hereinafter referred to as "DMEA");

di-amino bicyclo-octane (hereinafter referred to as "DABCO"); and

N,N-dimethyl cyclohexylamine (hereinafter referred to as "DMCHA").

- 5 Suitable organometallic tin-based compounds may be selected from the following:

stannous octoate, dibutyl tin dilaurate, dibutyl tin mercaptide, dibutyl tin thiocarboxylate, and dioctyl tin thiocarboxylate.

- 10 Other suitable organometallic compounds may include ferric acetylacetonate.

Suitable alkali metal salts may be selected from the following:

calcium carbonate, salts of carbonic acid, and salts of acetic acid.

15

One supplier of the above catalysts (as mixtures or single compounds) is Air Products, South Africa, and the catalysts may be identified inter alia by the following trade marks/brands and/or acronyms, respectively:

<u>CHEMICAL BRAND NAME</u>	<u>ACTIVE INGREDIENT(S)</u>
DMEA	2-Dimethyl ethanolamine
DABCO	Di-amino bicyclo-octane
POLYCAT 8 (DMCIIA)	<u>N,N-dimethyl cyclohexylamine</u>
DABCO R-8020	Triethylenediamine and Dimethylethanolamine
DABCO DC-1	Tin and amine complexes
DABCO DC-2	Tin and amine complexes
DABCO K-15	Potassium octate and diethylene glycol
DABCO TMR 2	Quaternary ammonium salt in ethylene glycol
THORCAT 401	Di-N-butyltindilaurate

When used with one or more of the above catalysts, the amount of MDI required to effect a suitable hastening of the curing process may preferably be in the range of about 0.1% to 1.9% on bone dry wood (hereinafter referred to as "BDW").

According to another aspect of the present invention, there is provided an additive for a binding mixture for use in manufacturing chipboard, the additive including an effective amount of MDI and a catalyst, each as indicated above, and as otherwise described herein.

According to a further aspect of the present invention, there is provided a process for manufacturing chipboard, the process including the steps of

adding to or using in a binding mixture consisting of urea-formaldehyde resin, an effective amount of MDI and a catalyst, each catalyst being as indicated above, or as otherwise herein described, or an additive, respectively, as herein described, and suitably mixing the binding mixture
5 with wood particles and/or chips.

DETAILED DESCRIPTION OF THE INVENTION

The invention will now be described in greater detail by way of non-
10 limiting example(s), with reference to the following:

1. LABORATORY TESTS

Laboratory tests were carried out by way of reasonable technical trials,
15 by personnel of the applicant's wholly owned subsidiary namely Sappi Timber Industries (Proprietary) Limited on wood particles and chips as used for manufacturing chipboard in the factories of the aforementioned company.

20 Using the aforementioned raw materials, binder mixtures of urea-formaldehyde resin as typically used in the aforementioned company's production facilities were applied to the aforementioned particle chips,

and compared with urea-formaldehyde mixtures including MDI, and further compared with mixtures of urea-formaldehyde with MDI and various catalysts as set out above.

5 The resulting mixtures of particles/chips and binder mixtures were pressed in a laboratory scale press to simulate plant production conditions, and curing times were measured. In the binding mixtures that did not include a catalyst, longer curing times were observed.

10 Details of the mixtures used and the corresponding results are set out in Table 1 hereafter:

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30

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TABLE 1

MIXTURE ADDED TO UF RESIN	INCREASE IN PRODUCTION SPEED (%)
<u>MDI + RELEASE AGENT</u> 1.4 % (on BDW) of MDI + 0.5 % (on UF solids) Internal Release and Wetting Agent	34.6
<u>MODIFIED MDI</u> (a) 1.4% (on BDW) MDI modified with PPG diol MDI pre-polymer (b) 1.4% (on BDW) MDI modified with PPG Triol MDI pre-polymer (c) 1.4% (on BDW) MDI modified with TDI (d) 1.4% (on BDW) MDI modified with PPG Triol TDI pre-polymer	44.3 44.3 44.3 42.4

Table 1 (Contd)

<u>CATALYSTS</u>	
(a) 1.4% (on BDW) MDI modified with PPG Triol MDI pre-polymer + 0.1% DABCO TMR 2 catalyst	45.8
(b) 1.4% (on BDW) MDI modified with PPG Triol MDI pre-polymer + 0.1% (on UF mass) DABCO K 15 catalyst	44.3
(c) 1.4% (on BDW) MDI modified with PPG Triol MDI pre-polymer + 0.1% (on UF mass) DABCO DC 2 catalyst	44.3
(d) 1.2% (on BDW) MDI modified with PPG Triol MDI pre-polymer + 0.1% (on UF mass) DMCHA catalyst	42.3
(e) (1.2% (on BDW) MDI modified with PPG Triol MDI pre-polymer + 0.1% (on UF mass) DABCO R8020 catalyst	42.3
(f) 1.2% (on BDW) MDI modified with PPG Triol MDI pre-polymer + 0.1% (on UF mass) Thorcat catalyst	40.0
(g) 1.4% (on BDW) MDI modified with Polyethylene glycol MDI pre-polymer + 0.1% (on UF mass) DABCO DC 2 catalyst	42.8
(h) 0.5% (on BDW) MDI + 1.1% (on UF mass) DMEA catalyst	33.3
(i) 0.5% (on BDW) water emulsified MDI + 1.1% (on UF mass) DMEA catalyst	33.3

In the aforementioned table, the brand name Daitolac R130 is a brand name for polyether polyol, which is sold by ICI (Imperial Chemical Industries). Similarly PPG diol MDI Pre-Polymer is the brand name for polypropylene glycol diol MDI pre-polymer and PPG triol TDI Pre-Polymer is the brand name for polypropylene glycol triol TDI pre-polymer.

The curing times for mixtures where typically 0.1% to 1.9% MDI and one or more of the abovementioned catalysts were added (on BDW), were the shortest.

10

The results of the above tests indicated that in all cases the binding mixtures with MDI and one or more catalysts resulted in a shorter curing period with the same or an increased binding strength.

15 It was also found that the use of amines as catalysts alone resulted in the least costly mixture and hence the most cost-effective process(es).

2. PRODUCTION-PLANT TRIALS

20 The aforementioned tests were repeated by way of reasonable technical trials, on two different production lines namely one using a single daylight press and the other using a continuous press. Details of the

mixtures used and the corresponding results are set out in Table 2 hereunder:

5 TABLE 2

MIXTURE ADDED TO UF RESIN	INCREASE IN PRODUCTION SPEED (%)
<u>CONTINUOUS PRESS</u>	
<u>WHITE RIVER FACTORY</u>	
0.5% (on BDW) waster emulsifiable MDI + 1.1% (on UF mass) + DMEA catalyst	14.3

The abovementioned production-line tests confirmed the aforementioned laboratory results, with improvements in or increased curing times of up to approximately 14 to 24%. The applicant believes that it may be possible to achieve greater improvements than the aforementioned.

It will therefore be seen from the aforementioned that a considerable improvement in curing times and hence in productivity may be possible by using the aforementioned invention. Such improvement also appears to be cost-effective relative to increased productivity and production on an industrial scale.

Although certain embodiments only of the invention have been described herein, it will be apparent to any person skilled in the art that other variations and/or modifications of the invention are possible. Such variations and/or modifications are therefore to be considered as falling
5 within the spirit and scope of the invention as claimed hereinafter.

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CLAIMS

1. A binding mixture for use in manufacturing chipboard, the mixture
5 including an effective amount of methyl di-isocyanate ("MDI") and
one or more polyurethane catalyst(s) being selected from the
following:
 - (a) one or more amine compound(s) including aliphatic and
aromatic tertiary amine derivatives of phenols, esters,
10 ethers, alkenes and/or alcohols; or
 - (b) one or more organometallic compounds of tin, bismuth,
zinc, iron, and/or alkali metal salt(s); or
 - (c) suitable mixtures of (a) and (b) above.
- 15 2. A binding mixture as claimed in claim 1, wherein the MDI is water-
emulsifiable.
3. A binding mixture as claimed in either claim 1 or claim 2, wherein
the or each polyurethane catalyst(s) is/are (a) delayed-action
20 catalyst(s).
4. A binding mixture as claimed in any one of the preceding claims,
wherein an effective amount of toluene di-isocyanate ("TDI") is
added to the MDI.

5. A binding mixture as claimed in any one of the preceding claims, wherein one or more internal wetting and release agents is/are added to the mixture.

5

6. A binding mixture as claimed in any one of the preceding claims, wherein a suitable amount of urea-formaldehyde resin is added.

10

7. A binding mixture as claimed in claim 6, wherein a suitable quantity of or more one polyol(s) is/are added.

8. A binding mixture as claimed in either claim 6 or claim 7, wherein the amount of formaldehyde in the urea-formaldehyde resin may be approximately 50% (in moles).

15

9. A binding mixture as claimed in any one of the preceding claims, wherein the or each suitable amine(s) or amine compound(s) are selected from the following:

2-dimethyl ethanolamine ("DMEA");

20 di-amino bicyclo-octane ("DABCO"); and

N,N-dimethyl cyclohexylamine ("DMCHA").

10. A binding mixture as claimed in any one of the preceding claims, wherein the or each suitable organometallic tin-based compound(s) is/are selected from the following:

Stannous octoate, dibutyl tin dilaurate, dibutyl tin mercaptide,
5 dibutyl tin thiocarboxylate, and dioctyl tin thiocarboxylate.

11. A binding mixture as claimed in any one of the preceding claims, wherein the suitable organometallic compounds include ferric acetylacetonate.

10

12. A binding mixture as claimed in any one of the preceding claims, wherein the suitable alkali metal salts are selected from the following:

calcium carbonate, salts of carbonic acid, and salts of acetic acid.

15

13. A binding mixture as claimed in any one of the preceding claims, wherein the amount of MDI required to effect a suitable hastening of the curing process is in the range of from about 0.1% to 1.9% of bone dry wood ("BDW").

20

14. A binding mixture substantially as herein described and/or exemplified.

15. An additive for a binding mixture for use in manufacturing chipboard, the additive including an effective amount of MDI and a catalyst, each catalyst being as claimed in any one of the preceding claims.
- 5
16. An additive for a binding mixture for use in making chipboard, substantially as herein described and/or exemplified.
17. A process for manufacturing chipboard, the process including the steps of adding to or using in a binding mixture an effective amount of MDI and a catalyst, each catalyst being as claimed in any one of claims 1 to 14, and suitably mixing the binding mixture with wood particles and/or chips.
- 10
18. A process for manufacturing chipboard, the process including the steps of adding to or using in a binding mixture an effective amount of an additive as claimed in either claim 15 or claim 16, and suitably mixing the binding mixture with wood particles and/or chips.
- 15
19. A process for manufacturing chipboard, substantially as herein described and/or exemplified.
- 20